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(54) Title: SILOXANE DRY CLEANING COMPOSITION AND PROCESS

(57) Abstract: A dry cleaning composition comprising a volatile siloxane and an organic surfactant and, optionally water, and a method for dry cleaning comprising contacting an article with a composition comprising a volatile siloxane and an organic surfactant.

## SILOXANE DRY CLEANING COMPOSITION AND PROCESS

## CROSS-REFERENCE TO RELATED APPLICATIONS

This application claims rights of priority from U.S. Provisional Patent Application Serial No. 60/184,108, filed February 22, 2000.

## TECHNICAL FIELD

The present invention is directed to a dry cleaning composition, more specifically, to a siloxane fluid based composition, for use in dry cleaning and to a dry cleaning process using the composition.

## BACKGROUND

Current dry cleaning technology uses perchloroethylene ("PERC") or  
5 petroleum-based materials as the cleaning solvent. PERC suffers from toxicity and odor issues. The petroleum-based products are not as effective as PERC in cleaning garments.

Cyclic siloxanes have been reported as spot cleaning solutions, see US 4,685,930, and as dry cleaning fluids in dry cleaning machines, see US  
10 5,942,007. Other patents disclose the use of silicone soaps in petroleum solvents, see JP 09299687, and the use of silicone surfactants in super critical carbon dioxide solutions has been reported, see, for example, US 5,676,705 and Chem. Mark. Rep., 15 Dec 1997, 252(24), p. 15. Non-volatile silicone oils have also been used as the cleaning solvent requiring removal by a second  
15 washing with perfluoroalkane to remove the silicone oil, see JP 06327888.

Numerous other patents have issued in which siloxanes or organomodified silicones have been present as addenda in PERC or petroleum based dry cleaning solvents, see, for example, WO 9401510; US 4911853; US 4005231; US 4065258.

There is a continued interest in providing an additive or additives to enhance the cleaning ability of silicone based dry cleaning solvents.

#### SUMMARY OF THE INVENTION

In a first aspect, the present invention is directed to a dry cleaning composition, comprising a volatile cyclic, linear or branched siloxane and one  
5 or more organic surfactants.

In a second aspect, the present invention is directed to a method for dry cleaning an article, comprising contacting the article with a composition comprising a cyclic, linear or branched siloxane and an organic surfactant which may be chosen from the classes of nonionic, cationic, anionic or  
10 amphoteric.

The process of the present invention exhibits improved performance, such as for example, removal of water soluble stains from the article, for example a garment, being cleaned. The process of the present invention also effectively removes most soluble stains, including oil stains and grease stains.

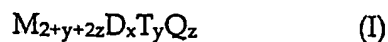
#### DETAILED DESCRIPTION OF THE INVENTION

15 In a preferred embodiment, the composition comprises, based on 100 parts by weight ("pbw") of the composition, from greater than 90 pbw to 99.99 pbw, more preferably from 92 pbw to 99.9 pbw and even more preferably from 95 pbw to 99.5 pbw of the siloxane and from 0.001 pbw to less than 10 pbw, more preferably from 0.01 pbw to 8 pbw and even more  
20 preferably from 0.1 pbw to 5 pbw of the surfactant. The composition optionally further comprises water, preferably from 0.01 pbw to 15 pbw, more preferably from 0.1 pbw to less than 12 pbw and even more preferably from 0.2 pbw to 10 pbw of water. Preferably, the composition does not include siloxane resins or crosslinking agents.

In a preferred embodiment, the water may be added as "free" water or may be delivered by an emulsion containing other components such as siloxanes, hydrocarbons, surfactants, or other suitable additives. If the water is delivered by an emulsion, the emulsion may be prepared by either  
 5 homogenization of the components or by mechanically stirring the mixture.

Compounds suitable as the linear or branched, volatile siloxane solvent of the present invention are those containing a polysiloxane structure that includes from 2 to 20 silicon atoms. Preferably, the linear or branched, volatile siloxanes are relatively volatile materials, having, for example, a  
 10 boiling of below about 300°C point at a pressure of 760 millimeters of mercury ("mm Hg").

In a preferred embodiment, the linear or branched, volatile siloxane comprises one or more compounds of the structural formula (I):



15 wherein:

M is  $R^1_3SiO_{1/2}$ ;

D is  $R^2R^3SiO_{2/2}$ ;

T is  $R^4SiO_{3/2}$ ;

and Q is  $SiO_{4/2}$

20  $R^1$ ,  $R^2$ ,  $R^3$  and  $R^4$  are each independently a monovalent hydrocarbon radical;  
 and

x and y are each integers, wherein  $0 \leq x \leq 10$  and  $0 \leq y \leq 10$  and  $0 \leq z \leq 10$ .

Suitable monovalent hydrocarbon groups include acyclic hydrocarbon radicals, monovalent alicyclic hydrocarbon radicals, monovalent and

aromatic or fluoro containing hydrocarbon radicals. Preferred monovalent hydrocarbon radicals are monovalent alkyl radicals, monovalent aryl radicals and monovalent aralkyl radicals.

As used herein, the term "(C<sub>1</sub>-C<sub>6</sub>)alkyl" means a linear or branched alkyl group containing from 1 to 6 carbons per group, such as, for example, methyl, ethyl, propyl, iso-propyl, n-butyl, iso-butyl, sec-butyl, tert-butyl, pentyl, hexyl, preferably methyl.

As used herein, the term "aryl" means a monovalent unsaturated hydrocarbon ring system containing one or more aromatic or fluoro containing rings per group, which may optionally be substituted on the one or more aromatic or fluoro containing rings, preferably with one or more (C<sub>1</sub>-C<sub>6</sub>)alkyl groups and which, in the case of two or more rings, may be fused rings, including, for example, phenyl, 2,4,6-trimethylphenyl, 2-isopropylmethylphenyl, 1-pentalenyl, naphthyl, anthryl, preferably phenyl.

As used herein, the term "aralkyl" means an aryl derivative of an alkyl group, preferably a (C<sub>2</sub>-C<sub>6</sub>)alkyl group, wherein the alkyl portion of the aryl derivative may, optionally, be interrupted by an oxygen atom, such as, for example, phenylethyl, phenylpropyl, 2-(1-naphthyl)ethyl, preferably phenylpropyl, phenoxypropyl, biphenyloxypropyl.

In a preferred embodiment, the monovalent hydrocarbon radical is a monovalent (C<sub>1</sub>-C<sub>6</sub>)alkyl radical, most preferably, methyl.

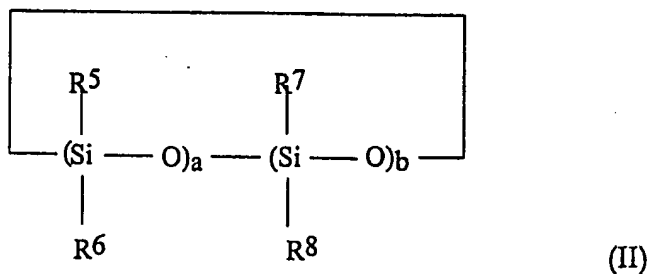
In a preferred embodiment, the linear or branched, volatile siloxane comprises one or more of, hexamethyldisiloxane, octamethyltrisiloxane, decamethyltetrasiloxane, dodecamethylpentasiloxane, tetradecamethylhexasiloxane or hexadecamethylheptasiloxane or methyltris(trimethylsiloxy)silane. In a more highly preferred embodiment, the linear or branched, volatile siloxane of the present invention comprises

octamethyltrisiloxane, decamethyltetrasiloxane, or dodecamethylpentasiloxane or methyltris(trimethylsiloxy)silane. In a highly preferred embodiment, the siloxane component of the composition of the present invention consists essentially of decamethyltetrasiloxane.

5            Suitable linear or branched volatile siloxanes are made by known methods, such as, for example, hydrolysis and condensation of one or more of tetrachlorosilane, methyltrichlorosilane, dimethyldichlorosilane, trimethylchlorosilane, or by isolation of the desired fraction of an equilibrate mixture of hexamethyldisiloxane and octamethylcyclotetrasiloxane or the like  
10            and are commercially available.

              Compounds suitable as the cyclic siloxane component of the present invention are those containing a polysiloxane ring structure that includes from 2 to 20 silicon atoms in the ring. Preferably, the linear, volatile siloxanes and cyclic siloxanes are relatively volatile materials, having, for example, a  
15            boiling point of below about 300°C at a pressure of 760 millimeters of mercury ("mm Hg").

              In a preferred embodiment, the cyclic siloxane component comprises one or more compounds of the structural formula (II):



20            wherein:

R<sup>5</sup>, R<sup>6</sup>, R<sup>7</sup> and R<sup>8</sup> are each independently a monovalent hydrocarbon group;  
and

a and b are each integers wherein  $0 \leq a \leq 10$  and  $0 \leq b \leq 10$ , provided that  $3 \leq (a + b) \leq 10$ .

In a preferred embodiment, the cyclic siloxane comprises one or more of, octamethylcyclotetrasiloxane, decamethylcyclopentasiloxane, 5 dodecamethylcyclohexasiloxane, tetradecamethylcycloheptasiloxane. In a more highly preferred embodiment, the cyclic siloxane of the present invention comprises octamethylcyclotetrasiloxane or decamethylcyclopentasiloxane. In a highly preferred embodiment, the cyclic siloxane component of the composition of the present invention consists 10 essentially of decamethylcyclopentasiloxane.

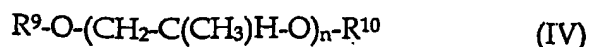
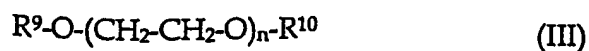
Suitable cyclic siloxanes are made by known methods, such as, for example, hydrolysis and condensation of dimethyldichlorosilane and are commercially available.

The organic surfactant of the present invention comprises one or more 15 surfactants selected from nonionic, cationic, anionic and amphoteric surfactants. In another embodiment, the organic surfactant comprises a mixture of two or more surfactants of the same or different classes, as long as they are compatible, such as, for example, a mixture of two or more nonionic, cationic, anionic or amphoteric surfactants, a mixture of nonionic and cationic 20 surfactants, a mixture of nonionic and anionic surfactants, a mixture of nonionic and amphoteric surfactants, a mixture of cationic and anionic surfactants, a mixture of cationic and amphoteric surfactants, a mixture of anionic and amphoteric surfactants, a mixture of nonionic, cationic and anionic surfactants, a mixture of nonionic, anionic and amphoteric 25 surfactants, a mixture of cationic anionic and amphoteric surfactants, or a mixture of nonionic, cationic, anionic and amphoteric surfactants.

Compounds suitable for use as the nonionic surfactant of the present invention are those that carry no discrete charge when dissolved in aqueous media. Nonionic surfactants are generally known in the art and include, for example, alkanol amides (such as, for example, coco, lauric, oleic and stearic monoethanolamides, diethanolamides and monoisopropanolamides), amine  
5 oxides (such as, for example, polyoxyethylene ethanolamides and polyoxyethylene propanolamides), polyalkylene oxide block copolymers (such as, for example, poly(oxyethylene-co-oxypropylene)), ethoxylated alcohols, (such as, for example, isostearyl polyoxyethylene alcohol, lauryl, cetyl, stearyl, oleyl, tridecyl, trimethylnonyl, isodecyl, tridecyl),  
10 ethoxylated alkylphenols (such as, for example, nonylphenol), ethoxylated amines and ethoxylated amides, ethoxylated fatty acids, ethoxylated fatty esters and ethoxylated fatty oils (such as, for example, mono- and diesters of acids such as lauric, isostearic, pelargonic, oleic, coco, stearic, and ricinoleic, and oils  
15 such as castor oil and tall oil), fatty esters, fluorocarbon containing materials, glycerol esters (such as, for example, glycerol monostearate, glycerol monolaurate, glycerol dilaurate, glycerol monoricinoleate, and glycerol oleate), glycol esters (such as, for example, propylene glycol monostearate, ethylene glycol monostearate, ethylene glycol distearate, diethylene glycol monolaurate, diethylene glycol monolaurate, diethylene glycol monooleate,  
20 and diethylene glycol stearate), lanolin-based surfactants, monoglycerides, phosphate esters, polysaccharide ethers, propoxylated fatty acids, propoxylated alcohols, and propoxylated alkylphenols, protein-based organic surfactants, sorbitan-based surfactants (such as, for example, sorbitan oleate, sorbitan monolaurate, and sorbitan palmitate), sucrose esters and glucose  
25 esters, and thio- and mercapto-based surfactants.

In a preferred embodiment, one component of the present invention comprises one or more nonionic surfactants according to one or more of the structural formulas III and IV:

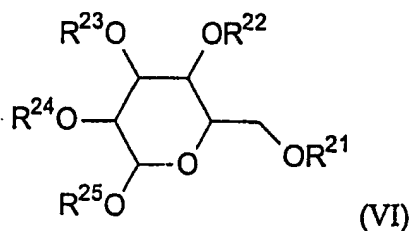
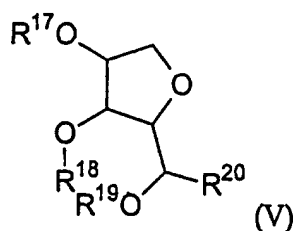




wherein:

$R^9$  is a monovalent hydrocarbon group of 1-30 carbons that may be  
 5 linear, cyclic, branched, unsaturated, aromatic or fluoro containing,  $R^{10}$  is  
 hydrogen or a monovalent hydrocarbon group of 1 to 30 carbons that may be  
 linear, cyclic, branched, unsaturated, aromatic or fluoro containing, and  $n$  is  
 from about 1 to about 100, more preferably from about 1 to about 40. In a  
 highly preferred embodiment,  $R^9$  contains from 2 to about 24 carbons, even  
 10 more preferably from 8 to 24 carbons,  $R^{10}$  is H and  $n$  is from about 2 to about  
 20.

In another preferred embodiment, one component of the present  
 invention comprises one or more nonionic surfactants that may be a sugar-  
 based surfactant according to one or more of the structural formulas V and  
 15 VI:

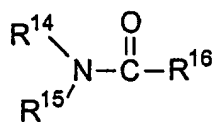


wherein:

each  $R^{17}$ ,  $R^{18}$ ,  $R^{19}$ ,  $R^{20}$ ,  $R^{21}$ ,  $R^{22}$ ,  $R^{23}$ ,  $R^{24}$  and  $R^{25}$  is independently H or a monovalent hydrocarbon group of 1 to 30 carbons that may be linear, cyclic, branched, an oxygenated alkane or other chalcogen containing group.

5 Chalcogens are herein specifically defined as oxygen, sulfur, selenium, tellurium and polonium. These surfactants may also be the open-chain analogs. In a preferred embodiment,  $R^{17}$ ,  $R^{18}$ ,  $R^{19}$ ,  $R^{20}$ ,  $R^{21}$ ,  $R^{22}$ ,  $R^{23}$ ,  $R^{24}$  and  $R^{25}$  are each H or a hydrocarbon group of 1 to 24 carbons, preferably a polyether or ester, even more preferably, one of  $R^{17}$  and  $R^{20}$  is a hydrocarbon of from 8  
10 to 24 carbons while the other is H or a hydrocarbon of from 1 to 4 carbons, such as  $-\text{CH}_2\text{OH}$  or  $-\text{CH}_2\text{CH}_3$ , and one of  $R^{21}$  and  $R^{25}$  is H or a hydrocarbon of from 8 to 24 carbons while the other is a hydrocarbon of from 1 to 4 carbons, such as  $-\text{CH}_2\text{OH}$  or  $-\text{CH}_2\text{CH}_3$ . In another preferred embodiment, the surfactant or surfactants are chosen from sucrose esters, glucose esters,  
15 monoglycerides, polysaccharide ethers and sorbitan-based surfactants.

In another preferred embodiment, one component of the present invention comprises one or more nonionic surfactants that may be an amine-based or phosphate ester-based surfactant according to one or more of the structural formulas VII and VIII:



(VII)

(VIII)

wherein:

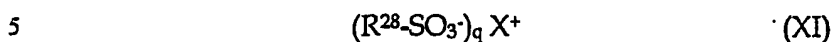
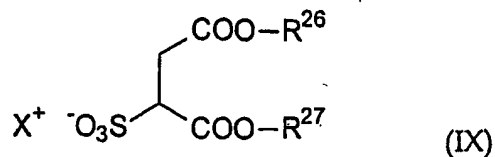
each  $R^{11}$ ,  $R^{12}$ ,  $R^{13}$ ,  $R^{14}$ ,  $R^{15}$ , and  $R^{16}$  is independently H or a monovalent hydrocarbon group of 1 to 30 carbons that may be linear, cyclic, branched,  
25 unsaturated, aromatic, fluoro containing, an oxygenated alkane or other

chalcogen containing group. In a preferred embodiment, two of R<sup>11</sup>, R<sup>12</sup> and R<sup>13</sup>, are H or hydrocarbon groups of 1 to 4 carbons, and one is a hydrocarbon group of from 8 to 24 carbons, and R<sup>14</sup> and R<sup>15</sup> are either H or hydrocarbon groups of from 1 to 4 carbons while R<sup>16</sup> is a hydrocarbon group of from 8 to 24 carbons, or R<sup>14</sup> and R<sup>15</sup> are hydrocarbon groups of from 8 to 24 carbons while R<sup>16</sup> is a hydrocarbon group of from 1 to 4 carbons. In a most preferred embodiment, the surfactant or surfactants are chosen from alkanol amides, amine oxides, ethoxylated amines, ethoxylated amides and phosphate esters.

Compounds suitable for use as the anionic surfactant of the present invention are those having polar, solubilizing groups such as carboxylate, sulfonate, sulfate and phosphate. Anionic surfactants are generally known in the art and include, for example, alkyl aryl sulfonates (such as, for example, alkylbenzenesulfonates), alkyl aryl sulfonic acids (such as, for example, sodium and ammonium salts of toluene-, xylene- and isopropylbenzenesulfonic acids), sulfonated amines and sulfonated amides (such as, for example, amidosulfonates), carboxylated alcohols and carboxylated alkylphenol ethoxylates, diphenyl sulfonates, fatty esters, isethionates, lignin-based surfactants, olefin sulfonates (such as, for example, RCH=CHSO<sub>3</sub>Na, where R is C<sub>10</sub>-C<sub>16</sub>), phosphorous-based surfactants, protein based surfactants, sarcosine-based surfactants (such as, for example, N-acylsarcosinates such as sodium N-lauroylsarcosinate), sulfates and sulfonates of oils and/or fatty acids, sulfates and sulfonates of ethoxylated alkylphenols, sulfates of alcohols, sulfates of ethoxylated alcohols, sulfates of fatty esters, sulfates of aromatic or fluoro containing compounds, sulfosuccinamates, sulfosuccinates (such as, for example, diamyl-, dioctyl- and diisobutylsulfosuccinates), taurates, and sulfonic acids.

In a preferred embodiment, one component of the present invention comprises one or more anionic surfactants that may be a sulfosuccinate,

sulfate, sulfonate, carboxylate, or phosphorous containing surfactant according to one or more of the structural formulas IX to XIII:



wherein:

each  $\text{R}^{26}$ ,  $\text{R}^{27}$ ,  $\text{R}^{28}$ ,  $\text{R}^{29}$  and  $\text{R}^{30}$  is independently a monovalent hydrocarbon group of 1 to 30 carbons that may be linear, cyclic, branched, unsaturated, aromatic, fluoro containing, an oxygenated alkane or other chalcogen containing radical, and X is H or an alkali metal, alkaline earth element or a chalcogen containing counterion or other suitable cation that does not unduly interfere with the functioning of the molecule as a surfactant where the subscript q is the valence or oxidation state of the cation X. In a preferred embodiment,  $\text{R}^{26}$  and  $\text{R}^{27}$  are linear hydrocarbon groups of from 4 to 20 carbons, more preferably 6 to 13 carbons,  $\text{R}^{28}$  is a hydrocarbon group of from 6 to 20 carbons, more preferably from 8 to 16 carbons, and  $\text{R}^{29}$  is a hydrocarbon group of from 8 to 26 carbons, more preferably from 10 to 20 carbons, and  $\text{R}^{30}$  is a hydrocarbon of from 8 to 30 carbons.

Compounds suitable for use as the cationic surfactant of the present invention are those having a positive charge when dissolved in aqueous media, which resides on an amino or quaternary nitrogen. Cationic

surfactants are generally known in the art and include, for example, amine acetates, amines (such as, for example, oxygen-free amines such as monoalkylamines, dialkylamines and N-alkyltrimethylene diamines, and oxygen-containing amines such as amine oxides, ethoxylated alkylamines, 1-  
 5 (2-hydroxyethyl)-2-imidazolines, and alkoxyates of ethylenediamine), and quaternary ammonium salts (such as, for example, dialkyldimethylammonium salts, alkylbenzyltrimethylammonium chlorides, alkyltrimethylammonium salts and alkylpyridium halides), and quaternary ammonium esters (such as, for example, diethyl ester dimethyl ammonium  
 10 chloride).

In a preferred embodiment, one component of the present invention comprises one or more cationic surfactants that may be a quaternary amine-based surfactant according to the structural formula XIV:



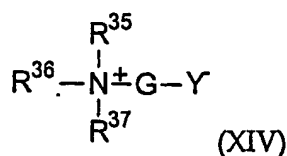
15 wherein:

each  $R^{31}$ ,  $R^{32}$ ,  $R^{33}$ , and  $R^{34}$  is independently H or a monovalent hydrocarbon group of 1 to 30 carbons that may be linear, cyclic, branched, unsaturated, aromatic, fluoro containing, an oxygenated alkane or other chalcogen containing group, and J is a suitable anion having an oxidation state or  
 20 valence p that does not unduly interfere with the functioning of the molecule as a surfactant. In a preferred embodiment,  $R^{31}$  and  $R^{32}$  are hydrocarbon groups of from 1 to 4 carbons, more preferably, methyl, and  $R^{33}$  and  $R^{34}$  are hydrocarbon groups of from 6 to 30 carbons, more preferably from 8 to 24 carbons.

25 Compounds suitable for use as the amphoteric surfactant of the present invention are those containing both an acidic and basic hydrophilic group. Amphoteric surfactants are compatible with anionic and cationic surfactants.

Amphoteric surfactants are generally known in the art and include, for example, betaine derivatives such as alkylbetaines and amidopropylbetaines, block copolymers, imidazolines and lecithins.

In a preferred embodiment, one component of the present invention comprises one or more amphoteric surfactants according to the structural formula XV:



wherein:

each  $\text{R}^{35}$ ,  $\text{R}^{36}$  and  $\text{R}^{37}$  is independently H or a monovalent hydrocarbon group of 1 to 30 carbons that may be linear, cyclic, branched, unsaturated, aromatic, fluoro containing, an oxygenated alkane or other chalcogen containing group, G is a divalent spacer group, and Y is a carboxylate, sulfonate, sulfate, phosphonate or other similar group. In a preferred embodiment,  $\text{R}^{35}$ , is a hydrocarbon of from 1 to 4 carbons, and  $\text{R}^{36}$  and  $\text{R}^{37}$  are hydrocarbons of from 6 to 24 carbons.

Surfactants are known in the art and are commercially available under many trade names from many sources, such as for example, Akzo Chemical Co., Calgene Chemical Inc., Emkay Chemical Co, Hercules, Inc., ICI Americas Inc., Lonza, Inc., Rhone Poulenc, Inc., Union Carbide Corp. and Witco Corp.

In a preferred embodiment, the dry cleaning composition of the present invention further comprises a minor amount, preferably, less than 50 pbw per 100 pbw of the composition, more preferably, less than 10 pbw per 100 pbw of the composition, of one or more non-siloxane fluids. Suitable non-siloxane fluids include aqueous fluids, such as, for example, water, and

organic fluids, for example, hydrocarbon fluids and halogenated hydrocarbon fluids.

5 An article, such as for example, a textile or leather article, typically, a garment, is dry cleaned by contacting the article with the composition of the present invention. In a preferred embodiment, the articles to be cleaned include textiles made from natural fibers, such as for example, cotton, wool, linen and hemp, from synthetic fibers, such as, for example, polyester fibers, polyamide fibers, polypropylene fibers and elastomeric fibers, from blends of natural and synthetic fibers, from natural or synthetic leather or natural or  
10 synthetic fur.

The article and dry cleaning composition are then separated, by, for example, one or more of draining and centrifugation. In a preferred embodiment, separation of the article and dry cleaning composition is followed by the application of heat, preferably, heating to a temperature of  
15 from 15°C to 120°C, preferably from 20°C to 100°C, or reduced pressure, preferably, a pressure of from 1 mm Hg to 750 mm Hg, or by application of both heat and reduced pressure, to the article.

Testing for water soluble stain removal was accomplished using fabric swatches supplied by the International Fabricare Institute ("IFI") (Silver  
20 Spring, MD) that contained a water soluble dye. The color change of a swatch of this material was measured by a Minolta CR-300® Colorimeter using the Hunter Color Number difference calculations. The larger the change in Hunter Color Number ( $\Delta E$ ), the greater the dye removal and the more efficient the cleaning.

25 The following examples are to illustrate the invention and are not to be construed as limiting the claims.

## EXAMPLES

Testing procedure: Circular swatches (from IFI) containing a water soluble dye were measured by the colorimeter, and the initial color values for L, a and b (as defined by the Hunter Color Numbers) were recorded. The fabric swatches were then placed in vials containing the cleaning composition of the present invention, and the vial was shaken for 10 minutes at ambient temperature. The fabric swatch was removed and allowed to drip dry for 2 to 5 seconds, then placed on absorbent toweling and allowed to air dry for 16 to 24 hours. A second reading of each fabric swatch was taken and the color difference ( $\Delta E$ ) was determined using the following formula:

$$\Delta E = [(L_1 - L_2)^2 + (a_1 - a_2)^2 + (b_1 - b_2)^2]^{1/2}$$

This color difference represents the relative amount of cleaning, with the higher  $\Delta E$  indicative of better cleaning performance.

## Example 1 - Nonionic surfactants [ethoxylated alcohols]

A cleaning composition according to the present invention containing a cyclic siloxane ( $D_5$ ) and one or more nonionic surfactants was made. Fabric swatches were cleaned using the above procedure, and the color difference was measured to determine the effectiveness of the cleaning composition. A solution of cyclic siloxane ( $D_5$ ) without a surfactant was used as a control.

Nonionic surfactants used in the example are those represented by formula III above, where  $R^9$  and  $n$  are as described in Table 1, and  $R^{10}$  is H.



Table 1 - Ethoxylated Alcohols

Exp. No.	R <sup>9</sup>	n	pbw	ΔE
1	C <sub>4</sub>	1	1	1.9
2	C <sub>4</sub>	1	5	2.7
3	C <sub>4</sub>	2	1	3.2
4	C <sub>4</sub>	2	5	3.2
5	C <sub>12-15</sub>	3	1	38.8
6	C <sub>12-15</sub>	3	5	41.1
7	C <sub>12-13</sub>	9	1	37.8
8	C <sub>12-13</sub>	9	5	38.7
9	C <sub>12-13</sub>	6.5	1	39.1
10	C <sub>12-13</sub>	6.5	5	38.7
11	C <sub>14-15</sub>	7	1	2.0 [18.7]
12	C <sub>14-15</sub>	7	5	39.0 [33.7]
13	C <sub>12-13</sub> / C <sub>4</sub>	6.5/1	5 (50/50)	41.5
14	C <sub>12-13</sub> / C <sub>4</sub>	9/1	5 (50/50)	42.9
15	C <sub>12-15</sub> / C <sub>4</sub>	3/1	5 (50/50)	13.8
16	C <sub>14-15</sub> / C <sub>4</sub>	7/1	5 (50/50)	41.1
17	C <sub>12</sub>	4	1	35.8
18	C <sub>12</sub>	4	5	40.7
19	C <sub>12</sub>	23	1	0.9
20	C <sub>12</sub>	23	5	1.3
21	C <sub>16</sub>	2	1	4.6
22	C <sub>16</sub>	2	5	2.0
23	C <sub>18</sub>	2	1	2.6
24	C <sub>18</sub>	2	5	19.0
25	C <sub>18</sub>	10	1	2.4
26	C <sub>18</sub>	10	5	23.4
27	C <sub>18</sub>	20	1	4.0
28	C <sub>18</sub>	20	5	22.8
29	C <sub>12</sub> / C <sub>4</sub>	4 /1	5 (50/50)	41.1
39	C <sub>12</sub> / C <sub>4</sub>	23/1	5 (50/50)	1.6
31	C <sub>16</sub> / C <sub>4</sub>	2/1	5 (50/50)	3.7
32	C <sub>18</sub> / C <sub>4</sub>	2/1	5 (50/50)	11.4
33	C <sub>18</sub> / C <sub>4</sub>	10/1	5 (50/50)	21.1
34	C <sub>18</sub> / C <sub>4</sub>	20/1	5 (50/50)	34.4
Control 1	--	--	0	1.9

Table 1 shows that nonionic surfactants enhance the cleaning and dye removal of the base cyclic siloxane (D<sub>5</sub>) solvent.

### Example 2 - Anionic Surfactants

A cleaning composition according to the present invention containing a cyclic siloxane (D<sub>5</sub>) and one or more anionic surfactants was made. Fabric swatches were cleaned using the above procedure, and the color difference was measured to determine the effectiveness of the cleaning composition. A solution of cyclic siloxane (D<sub>5</sub>) without a surfactant was used as a control.

Table 2 - Sulfosuccinates

Exp.	Designation*	mixture	pbw	$\Delta E$
35	Aerosol TR	--	1	2.8
36	Aerosol TR	--	5	6.5
37	Aerosol OT	--	1	1.6
38	Aerosol OT	--	5	2.3
39	Aerosol GPG	--	1	3.0
40	Aerosol GPG	--	5	3.0
41	Aerosol TR/OT	50/50	1	1.5
42	Aerosol TR/OT	50/50	5	2.5
43	Aerosol TR/GPG	50/50	1	6.9
44	Aerosol TR/GPG	50/50	5	16.9
45	Aerosol OT/GPG	50/50	1	4.6
46	Aerosol OT/GPG	50/50	5	6.7
Control 2	--	--	0	1.9

\*Commercially available from Cytek Industries

Table 2 shows that the anionic sulfosuccinate surfactants enhanced the water soluble dye removal of the base cyclic siloxane (D<sub>5</sub>) solvent. (Surfactant TR is a solution in 20% ethanol and 10% water; GPG is a solution in 8% ethanol and 22% water.)

### Example 3 - Cationic and Anionic Surfactants

A cleaning composition according to the present invention containing a cyclic siloxane (D<sub>5</sub>) and one or more anionic and cationic surfactants was made. Fabric swatches were cleaned using the above procedure, and the color

difference was measured to determine the effectiveness of the cleaning composition. A solution of cyclic siloxane (D<sub>5</sub>) without a surfactant was used as a control.

Table 3 - Ionic Surfactants (Cationic and Anionic)

Exp	Type	R	pbw	ΔE
47	R-SO <sub>4</sub> -Na <sup>+</sup>	C <sub>14-16</sub> alkene	5	11.2
48	R <sub>2</sub> Me <sub>2</sub> N <sup>+</sup> Cl <sup>-</sup>	C <sub>12</sub>	1	41.5
49	R <sub>2</sub> Me <sub>2</sub> N <sup>+</sup> Cl <sup>-</sup>	C <sub>12</sub>	5	41.2
50	DDBSA	--	1	51.5
51	DDBSA	--	5	50.4
52	R-PhO-(EO) <sub>3</sub> - OSO <sub>3</sub> -Na <sup>+</sup>	C <sub>12</sub>	1	6.2
53	R-PhO-(EO) <sub>3</sub> - OSO <sub>3</sub> -Na <sup>+</sup>	C <sub>12</sub>	5	5.3
54	R-SO <sub>4</sub> -Na <sup>+</sup>	C <sub>12</sub>	1	2.7
55	R-SO <sub>4</sub> -Na <sup>+</sup>	C <sub>12</sub>	5	3.4
Control 3	--	--	0	1.9

5 Table 3 shows that the ionic surfactants enhanced the water soluble dye removal of the base cyclic siloxane (D<sub>5</sub>) solvent. (R<sub>2</sub>Me<sub>2</sub>N<sup>+</sup>Cl<sup>-</sup> came as a solution in water.)

#### Example 4 - Nonionic surfactants with water

10 A cleaning composition according to the present invention containing a cyclic siloxane (D<sub>5</sub>), water and a nonionic surfactant was made. Fabric swatches were cleaned using the above procedure, and the color difference was measured to determine the effectiveness of the cleaning composition. A solution of cyclic siloxane (D<sub>5</sub>) without a surfactant was used as a control. Nonionic surfactants used in the example are those represented by formula III above, where R<sup>9</sup> and n are as described in Table 4, and R<sup>10</sup> is H.

Table 4 - Nonionic Surfactants

Exp. No.	R <sup>9</sup>	n	pbw solvent	pbw surfactant	pbw water	$\Delta E$
1	C <sub>12-13</sub>	6.5	95	4	1	39.5
2	C <sub>12-13</sub>	6.5	95	1	4	33.1
3	C <sub>12-13</sub>	6.5	98	1	1	13.8
4	C <sub>12-13</sub>	9	95	4	1	34.9
5	C <sub>12-13</sub>	9	95	1	4	38.3
6	C <sub>12-13</sub>	9	98	1	1	25.5
7	C <sub>12-15</sub>	3	95	4	1	7.7
8	C <sub>12-15</sub>	3	95	1	4	38.3
9	C <sub>12-15</sub>	3	98	1	1	38.9
10	C <sub>14-15</sub>	7	95	4	1	34.5
11	C <sub>14-15</sub>	7	95	1	4	36.4
12	C <sub>14-15</sub>	7	98	1	1	7.9
13	C <sub>12</sub>	4	95	4	1	17.9
14	C <sub>12</sub>	4	95	1	4	32.3
15	C <sub>12</sub>	4	98	1	1	37.4
16	C <sub>12</sub>	23	95	4	1	24.5
17	C <sub>12</sub>	23	95	1	4	34.2
18	C <sub>12</sub>	23	98	1	1	1.5
19	C <sub>16</sub>	20	95	4	1	25.7
20	C <sub>16</sub>	20	95	1	4	11.8
21	C <sub>16</sub>	20	98	1	1	17.4
22	C <sub>18</sub>	2	95	4	1	8.5
23	C <sub>18</sub>	2	95	1	4	7.9
24	C <sub>18</sub>	2	98	1	1	5.5
25	C <sub>18</sub>	10	95	4	1	16.8
26	C <sub>18</sub>	10	95	1	4	6.2
27	C <sub>18</sub>	10	98	1	1	3.7
28	C <sub>18</sub>	20	95	4	1	13.6
29	C <sub>18</sub>	20	95	1	4	28.4
30	C <sub>18</sub>	20	98	1	1	5.3
31	C <sub>4</sub>	1	95	4	1	6.2
32	C <sub>4</sub>	1	95	1	4	11.7
33	C <sub>4</sub>	1	98	1	1	1.0
34	C <sub>4</sub>	2	95	4	1	33.9
35	C <sub>4</sub>	2	95	1	4	34.1
36	C <sub>4</sub>	2	98	1	1	38.7
37	C <sub>11-14</sub>	12	95	4	1	24.1
38	C <sub>11-14</sub>	12	95	1	4	33.1
39	C <sub>11-14</sub>	12	98	1	1	10.2
Control 4	-	-	99	0	1	2.2
Control 5	-	-	96	0	4	9.5

Table 4A - Nonionic Surfactants (Commercially Available)

Exp.	Surfactant Trade Name	n	pbw solvent	pbw surfactant	pbw water	$\Delta E$
40	Triton X-405	40	95	4	1	37.7
41	Triton X-405	40	95	1	4	25.5
42	Triton X-405	40	98	1	1	15.9
43	Igepal CA-520	5	95	4	1	4.4
44	Igepal CA-520	5	95	1	4	10.0
45	Igepal CA-520	5	98	1	1	2.3
46	Igepal CO-850	20	95	4	1	4.0
47	Igepal CO-850	20	95	1	4	2.6
48	Igepal CO-850	20	98	1	1	16.2
49	Span-80	-	95	4	1	3.7
50	Span-80	-	95	1	4	2.4
51	Span-80	-	98	1	1	5.2
Control 1	-	-	99	0	1	2.2
Control 2	-	-	96	0	4	9.5

Tables 4 and 4A show that nonionic surfactants in the presence of water enhance the cleaning and dye removal of the base cyclic siloxane (D<sub>5</sub>) solvent.

#### Example 5 - Ionic Surfactants

5 A cleaning composition according to the present invention containing a cyclic siloxane (D<sub>5</sub>), water and an ionic surfactant was made. Fabric swatches were cleaned using the above procedure, and the color difference was measured to determine the effectiveness of the cleaning composition. A solution of cyclic siloxane (D<sub>5</sub>) and water without a surfactant was used as a control.

Table 5 – Ionic Surfactants

Exp	Surfactant Trade Name	pbw solvent	pbw surfactant	pbw Water	$\Delta E$
52	Aerosol OT	95	4	1	6.9
53	Aerosol OT	95	1	4	20.3
54	Aerosol OT	98	1	1	7.5
55	Triton X-200	95	4	1	4.0
56	Triton X-200	95	1	4	36.0
57	Triton X-200	98	1	1	3.3
58	Vari-Soft 300	95	4	1	40.3
59	Vari-Soft 300	95	1	4	38.4
60	Vari-Soft 300	98	1	1	35.9
61	Bio-Soft D-62	95	4	1	2.9
62	Bio-Soft D-62	95	1	4	28.4
63	Bio-Soft D-62	98	1	1	14.3
64	Ethoquad C/25	95	4	1	35.2
65	Ethoquad C/25	95	1	4	34.3
66	Ethoquad C/25	98	1	1	26.3
67	Span-80	95	4	1	3.7
68	Span-80	95	1	4	2.4
69	Span-80	98	1	1	5.2
70	Glucopon 225*	95	1	4	4.7
71	Glucopon 225	95	4	1	31.2
72	Glucopon 225	98	1	1	5.8
73	Glucopon 225	99	1	-	10.8
74	Glucopon 425**	95	1	4	32.5
75	Glucopon 425	95	4	1	36.2
76	Glucopon 425	98	1	1	19.0
77	Glucopon 425	99	1	-	3.9
78	Glucopon 600**	95	1	4	4.3
79	Glucopon 600	95	4	1	27.9
80	Glucopon 600	98	1	1	4.7
81	Glucopon 600	99	1	-	9.3
82	Alkamide S-280	95	5	-	8.4
83	Alkamide S-280	99	1	-	1.7
84	Alkamide S-280	98	1	1	2.0
85	Alkamide CME	95	5	-	7.6
86	Alkamide CME	99	1	-	2.4
87	Alkamide CME	98	1	1	6.6
Control 6	-	99	0	1	2.2
Control 7	-	96	0	4	9.5

\*30% water; \*\*50% water

Table 5 shows that the ionic surfactants in the presence of water enhanced the water soluble dye removal of the base cyclic siloxane (D<sub>5</sub>) solvent.

Table 6 - Ionic surfactants with and without water

Exp	pbw solvent	Surfactant	pbw surfactant	pbw Water	$\Delta E$
88	14.25	Cocoa/oleamidopropyl betaine (30% in water)	0.75	-	14.39
89	14.85	Cocoa/oleamidopropyl betaine (30% in water)	0.15	-	16.76
90	14.7	Cocoa/oleamidopropyl betaine (30% in water)	0.15	0.15	32.02
91	14.25	Cocomidopropyl betaine (29% in water)	0.6	-	31.20
92	14.85	Cocomidopropyl betaine (29% in water)	0.15	-	7.11
93	14.7	Cocomidopropyl betaine (29% in water)	0.15	0.15	29.80
94	14.25	Stearic acid monoethanolamide	0.6	-	8.37
95	14.85	Stearic acid monoethanolamide	0.15	-	1.72
96	14.7	Stearic acid monoethanolamide	0.15	0.15	1.96
97	14.25	Amphoteric surfactant (50% in water)	0.6	-	33.76
98	14.85	Amphoteric surfactant (50% in water)	0.15	-	24.95
99	14.7	Amphoteric surfactant (50% in water)	0.15	0.15	32.09
100	14.25	Coconut fatty acid monoethanolamide	0.6	-	7.61
101	14.85	Coconut fatty acid monoethanolamide	0.15	-	2.40
102	14.7	Coconut fatty acid monoethanolamide	0.15	0.15	6.59
103	14.25	1,2-hexanediol	0.6	-	3.86
104	14.85	1,2-hexanediol	0.15	-	21.40
105	14.7	1,2-hexanediol	0.15	0.15	14.85
106	14.85	Di(ethyleneglycol)-2-ethylhexyl ether	0.15	-	8.03
107	14.25	Di(ethyleneglycol)-2-ethylhexyl ether	0.75	-	10.40
108	14.25	Di(ethyleneglycol)-2-ethylhexyl ether	0.6	0.15	9.85
109	14.25	Di(ethyleneglycol)-2-ethylhexyl ether	0.15	0.6	13.97
110	14.7	Di(ethyleneglycol)-2-ethylhexyl ether	0.15	0.15	22.73
111	14.85	Di(ethyleneglycol)hexyl ether	0.15	-	8.89
112	14.25	Di(ethyleneglycol)hexyl ether	0.75	-	9.13
113	14.25	Di(ethyleneglycol)hexyl ether	0.6	0.15	33.40
114	14.25	Di(ethyleneglycol)hexyl ether	0.15	0.6	16.64
115	14.7	Di(ethyleneglycol)hexyl ether	0.15	0.15	24.02
116	14.85	Didecyldimethylammonium bromide	0.15	-	16.55
117	14.25	Didecyldimethylammonium bromide	0.75	-	15.44
118	14.25	Didecyldimethylammonium bromide	0.6	0.15	4.78
119	14.25	Didecyldimethylammonium bromide	0.15	0.6	10.36
120	14.7	Didecyldimethylammonium bromide	0.15	0.15	10.88
121	14.85	Dihexadecyldimethylammonium bromide	0.15	-	12.53
122	14.25	Dihexadecyldimethylammonium bromide	0.75	-	12.15
123	14.25	Dihexadecyldimethylammonium bromide	0.6	0.15	8.73
124	14.25	Dihexadecyldimethylammonium bromide	0.15	0.6	9.56
125	14.7	Dihexadecyldimethylammonium bromide	0.15	0.15	9.45
126	14.85	Cetyltrimethylammonium bromide	0.15	-	13.03
127	14.25	Cetyltrimethylammonium bromide	0.75	-	14.79
128	14.25	Cetyltrimethylammonium bromide	0.6	0.15	12.25
129	14.25	Cetyltrimethylammonium bromide	0.15	0.6	38.27
130	14.7	Cetyltrimethylammonium bromide	0.15	0.15	10.39
131	14.85	1,2-butanediol	0.15	-	26.14
132	14.25	1,2-butanediol	0.75	-	33.45
133	14.7	1,2-butanediol	0.15	0.15	21.40
134	14.85	1,2-decanediol	0.15	-	11.26

135	14.25	1,2-decanediol	0.75	-	29.54
136	14.7	1,2-decanediol	0.15	0.15	11.55
137	14.85	1,2-hexanediol	0.15	-	10.01
138	14.25	1,2-hexanediol	0.75	-	28.56
139	14.7	1,2-hexanediol	0.15	0.15	32.51
140	14.85	1,6-hexanediol	0.15	-	7.47
141	14.25	1,6-hexanediol	0.75	-	5.16
142	14.7	1,6-hexanediol	0.15	0.15	31.78
143	14.85	1,10-decanediol	0.15	-	5.82
144	14.25	1,10-decanediol	0.75	-	1.22
145	14.7	1,10-decanediol	0.15	0.15	8.33

Table 7 shows the variations in R and x that were explored for these surfactants. Mixtures of materials within a class were also examined as seen in experiments 13-16 and 29-34. None of these surfactants were soluble in D5 in the ranges examined but some were only slightly hazy. As seen in Table 1, the surfactants with  $R=C_{12-15}$  and  $x=3-9$  repeat units gave the best cleaning.

Table 7 - Ethoxylated Alcohols.

Exp.	Surfactant	R	n	pbw	$\Delta E$
146	06383	$C_4$	1	1	1.9
147		$C_4$	1	5	2.7
148	2-(2-n-butoxy ethoxy)ethanol	$C_4$	2	1	3.2
149	067012	$C_4$	2	5	3.2
150	Neodol 25-3	$C_{12-15}$	3	1	38.8
151		$C_{12-15}$	3	5	41.9
152	Neodol 23-9	$C_{12-13}$	9	1	37.8
153		$C_{12-13}$	9	5	38.7
154	Neodol 23-6.5	$C_{12-13}$	6.5	1	39.1
155		$C_{12-13}$	6.5	5	38.6
156	Neodol 45-7	$C_{14-15}$	7	1	18.7
157		$C_{14-15}$	7	5	36.7
158		$C_{14-15}$	7	5	30.7
159		$C_{12-13}/C_4$	6.5/1	5 (50/50)	41.5
160		$C_{12-13}/C_4$	9/1	5 (50/50)	42.9
161		$C_{12-13}/C_4$	3/1	5 (50/50)	13.8
162		$C_{14-15}/C_4$	7/1	5 (50/50)	41.1
163	BRIJ 30	$C_{12}$	4	1	35.8
164	067220	$C_{12}$	4	5	40.7
165	BRIJ 35	$C_{12}$	23	1	0.9
166	067219	$C_{12}$	23	5	1.3
167	BRIJ 58	$C_{16}$	20	1	4.6
168		$C_{16}$	20	5	2.0
169	BRIJ 72	$C_{18}$	2	1	2.6
170	067263	$C_{18}$	2	5	19.0
171	BRIJ 76	$C_{18}$	10	1	2.4
172	067262	$C_{18}$	10	5	23.4
173	BRIJ 78	$C_{18}$	20	1	4.0
174		$C_{18}$	20	5	22.8
175		$C_{12}/C_4$	4/1	5 (50/50)	41.1
176		$C_{12}/C_4$	23/1	5 (50/50)	1.6
177		$C_{16}/C_4$	2/1	5 (50/50)	3.7
178		$C_{18}/C_4$	2/1	5 (50/50)	11.4
179		$C_{18}/C_4$	10/1	5 (50/50)	21.1
180		$C_{18}/C_4$	20/1	5 (50/50)	34.4
Control I		--	--	0	1.9



When similar compositions of 1 and D<sub>3</sub> with water were examined, again, the best cleaning was seen with R=C<sub>12-15</sub> and x=3-9 repeat units (Table 2).

Table 2. Ethoxylated Alcohols with Water.

Exp. No.	Surfactant	R	n	pbw solvent	pbw surfactant	pbw water	ΔE
35	Neodol 23-6.5	C <sub>12-13</sub>	6.5	95	4	1	39.5
36		C <sub>12-13</sub>	6.5	95	1	4	33.1
37		C <sub>12-13</sub>	6.5	98	1	1	13.8
38	Neodol 23-9	C <sub>12-13</sub>	9	95	4	1	34.9
39		C <sub>12-13</sub>	9	95	1	4	38.3
40		C <sub>12-13</sub>	9	98	1	1	25.5
41	Neodol 25-3	C <sub>12-15</sub>	3	95	4	1	7.7
42		C <sub>12-15</sub>	3	95	1	4	38.3
43		C <sub>12-15</sub>	3	98	1	1	38.9
44	Neodol 45-7	C <sub>14-15</sub>	7	95	4	1	34.5
45		C <sub>14-15</sub>	7	95	1	4	36.4
46		C <sub>14-15</sub>	7	98	1	1	7.9
47	BRIJ 30	C <sub>12</sub>	4	95	4	1	17.9
48		C <sub>12</sub>	4	95	1	4	32.3
49		C <sub>12</sub>	4	98	1	1	37.4
50	BRIJ 35	C <sub>12</sub>	23	95	4	1	24.5
51		C <sub>12</sub>	23	95	1	4	34.2
52		C <sub>12</sub>	23	98	1	1	1.5
53	BRIJ 58	C <sub>16</sub>	20	95	4	1	25.7
54		C <sub>16</sub>	20	95	1	4	11.8
55		C <sub>16</sub>	20	98	1	1	17.4
56	BRIJ 72	C <sub>18</sub>	2	95	4	1	8.5
57		C <sub>18</sub>	2	95	1	4	7.9
58		C <sub>18</sub>	2	98	1	1	5.5
59	BRIJ 76	C <sub>18</sub>	10	95	4	1	16.8
60		C <sub>18</sub>	10	95	1	4	6.2
61		C <sub>18</sub>	10	98	1	1	3.7
62	BRIJ 78	C <sub>18</sub>	20	95	4	1	13.6
63		C <sub>18</sub>	20	95	1	4	28.4
64		C <sub>18</sub>	20	98	1	1	5.3
65	06383	C <sub>4</sub>	1	95	4	1	6.2
66		C <sub>4</sub>	1	95	1	4	11.7
67		C <sub>4</sub>	1	98	1	1	1.0
68	067012	C <sub>4</sub>	2	95	4	1	33.9
69		C <sub>4</sub>	2	95	1	4	34.1
70		C <sub>4</sub>	2	98	1	1	38.7
71		C <sub>11-14</sub>	12	95	4	1	24.1
72		C <sub>11-14</sub>	12	95	1	4	33.1
73		C <sub>11-14</sub>	12	98	1	1	10.2
Control 2		-	-	95	0	1	5.3
Control 3		-	-	99	0	4	2.9
Control 4		-	-	99	0	5	2.6

Ethoxylated phenols, 2, were also explored (Table 3). The most effective mixtures included longer EO chains and lower amounts of water.

5

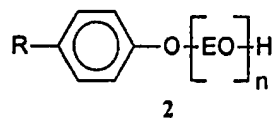


Table 3. Alkyl Phenol Surfactants.

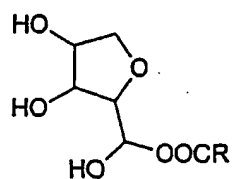
Exp.	Surfactant	R	n	pbw solvent	pbw surfactant	pbw water	$\Delta E$
74	Triton X-405	C <sub>9</sub>	40	95	4	1	37.7
75			40	95	1	4	25.5
76			40	98	1	1	15.9
77	Igepal CA-520	C <sub>12</sub>	5	95	4	1	4.4
78			5	95	1	4	10.0
79			5	98	1	1	2.3
80	Igepal CO-850	C <sub>9</sub>	20	95	4	1	4.0
81			20	95	1	4	2.6
82			20	98	1	1	16.2

Glycol ethers and diols were also examined as additives to enhance the cleaning ability of the silicone solvent as seen in Table 4.

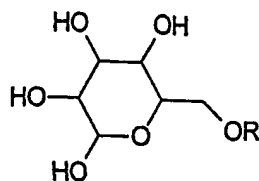
Table 4. Non-Ionic Ether and Diol Surfactants.

Exp. No.	Surfactant		pbw solvent	pbw surfactant	pbw water	$\Delta E$
83	AZ7989	di(ethyleneglycol)-2-ethylhexyl ether	99	1	-	8.03
84			95	5	-	10.40
85			95	4	1	9.85
86			95	1	4	13.97
87			98	1	1	22.73
88	AZ7988	di(ethyleneglycol)hexyl ether	99	1	-	8.89
89			95	5	-	9.13
90			95	4	1	33.40
91			95	1	4	16.64
92			98	1	1	24.02
93	AZ7997	1,2-butanediol	99	1	-	26.14
94			95	5	-	33.45
95			98	1	1	21.40
96	AZ7998	1,2-decanediol	99	1	-	11.26
97			95	5	-	29.54
98			98	1	1	11.55
99	AZ7995	1,2-hexanediol	99	1	-	10.01
100			95	5	-	28.56
101			95	5	-	15.7
102			98	1	1	32.51
103			99	1	-	21.40
104			98	1	1	14.85
105	AZ7996	1,6-hexanediol	99	1	-	7.47
106			95	5	-	5.16
107			98	1	1	31.78
108	AZ7999	1,10-decanediol	99	1	-	5.82
109			95	5	-	1.22
110			98	1	1	8.33

- 5 In the ether examples, optimal performance was seen with the addition of small amounts of water. The 1,2-diols were efficient at removing the dye at the 5% level, although significant cleaning was seen at 1% with water present.
- Table 5 shows the results from using sugar based surfactants and alkanol amides as water-based stain removers.



3  
Span



4  
Glucopon



5  
Alkamide

Table 5. Other Non-Ionic Surfactants.

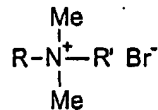
Exp. No.	Surfactant Trade Name	R	pbw solvent	pbw surfactant	pbw water	ΔE
111	Span-80	Oleic	95	4	1	3.7
112			95	1	4	2.4
113			98	1	1	5.2
114	Glucopon 225*	C <sub>8-10</sub>	95	1	4	4.7
115			95	4	1	31.2
116			98	1	1	5.8
117			99	1	-	10.8
118	Glucopon 425**	C <sub>8-16</sub>	95	1	4	32.5
119			95	4	1	36.2
120			98	1	1	19.0
121			99	1	-	3.9
122			95	5	-	19.6
123	Glucopon 600***	C <sub>10-16</sub>	95	1	4	4.3
124			95	4	1	27.9
125			98	1	1	4.7
126			99	1	-	9.3
127	Alkamide S-280	Stearic	95	5	-	8.37
128			99	1	-	1.72
129			98	1	1	1.96
130	Alkamide CME	Coconut	95	5	-	7.61
131			99	1	-	2.40
132			98	1	1	6.59

\*30% water, \*\* 50% water, \*\*\* 50% water

The sorbitan oleate, as Span 80, was fairly ineffective as a cleaning additive, but the 6-membered glucoside materials (Glucopans) exhibited good cleaning power at the 4% level with additional water. The two alkanol amides performed poorly as cleaning surfactants in these tests.

#### Cationics

The cationic surfactants tested were all quaternary ammonium salts of the type 6 below. As one can see, the quat salts were effective at the 1% level in all cases. Additional water was sometimes advantageous.



6

Table 6. Cationic Surfactants.

Exp. No.	Surfactant		pbw solvent	pbw surfactant	pbw water	$\Delta E$
133	AZ7987	Didecyldimethyl ammonium bromide	99	1	-	16.55
134			95	5	-	15.44
135			95	4	1	4.78
136			95	1	4	10.36
137			98	1	1	10.88
138	AZ7990	Dihexadecyldimethyl ammonium bromide	99	1	-	12.53
139			95	5	-	12.15
140			95	4	1	8.73
141			95	1	4	9.56
142			98	1	1	9.45
143	AZ7991	cetyltrimethylammonium bromide	99	1	-	13.03
144			95	5	-	14.79
145			95	4	1	12.25
146			95	1	4	38.27
147			98	1	1	10.39
148		Vari-Soft 300	95	4	1	40.3
149			95	1	4	38.4
150			98	1	1	35.9
151	06955	Ethoquad C/25	95	4	1	35.2
152			95	1	4	34.3
153			98	1	1	26.3
154			99	1	-	41.5
155			95	5	-	41.2

Vari-Soft 300: 30%  $(C_{16})Me_3N^+Cl^-$ ; Ethoquad C/25:  $C_{12-15}(Me)N((EO)_{25}H)_2^+Cl^-$

### Amphoterics

- 5 The amphoteric materials examined were of the betaine class as illustrated below (table7). These were quaternized glycine derivatives. All these materials were supplied as aqueous solutions and performed moderately well at high levels and even better at lower, 1% loading.

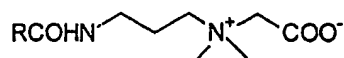


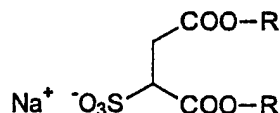
Table 7. Amphoteric Surfactants.

Exp. No.	Surfactant	R	pbw solvent	pbw surfactant	pbw water	?E
156	Mirataine COB*	Coco/oleo	95	5	-	14.39
157			99	1	-	16.76
158			98	1	1	32.02
159	Mirataine BET-C30**	Coco	95	5	-	31.20
160			99	1	-	7.11
161			98	1	1	29.80
162	Mirataine JC HA***		95	5	-	33.76
163			99	1	-	24.95
164			98	1	1	32.09

\*coca/oleamidopropyl betaine 30% in water, \*\* cocamidopropyl betaine 29% in water, \*\*\* amphoteric 50% in water

### Anionics

- 5 A wide variety of organic anionic surfactants are available in the forms of sulfosuccinates, sulfonates, phosphonates and the like. One set examined were the sulfosuccinates as shown in Table 8. Best results were seen with high levels of added water. One beneficial feature of the Aerosol OT was that it was soluble in D<sub>5</sub> to at least 5 weight percent.



10

Table 8. Sulfosuccinates.

Exp.	Surfactant Trade Name	R	mixture	pbw solvent	pbw surf	pbw water	ΔE
165	Aerosol TR	Tridecyl	-	99	1	-	2.8
166			-	95	5	-	6.5
167	Aerosol OT	Octyl	-	99	1	-	1.6
168			-	95	5	-	2.3
169			-	95	4	1	6.9
170			-	95	1	4	20.3
171			-	98	1	1	7.5
172	Aerosol GPG	Octyl	-	99	1	-	3.0
173			-	95	5	-	3.0
174	Aerosol TR/OT		50/50	99	1	-	1.5
175			50/50	95	5	-	2.5
176	Aerosol TR/GPG		50/50	99	1	-	6.9
177			50/50	95	5	-	16.9
178	Aerosol OT/GPG		50/50	99	1	-	4.6
179			50/50	95	5	-	6.7

Several phosphorous containing surfactants were tested as shown in Table 9. The ethoxylated phosphonates exhibited modest cleaning behavior while the lecithin-based surfactants did not remove the water soluble dye from the swatch.

15

Table 9. Phosphorous Containing Anionic Surfactants.

Exp. No.	Surfactant Trade Name	Pbw Solvent	pbw surfactant	pbw water	$\Delta E$
180	ATPHOS 3250	99	1	-	11.5
181		95	5	-	12.3
182		95	4	1	7.9
183		95	1	4	10.1
184		98	1	1	13.5
185	ATPHOS 3226	99	1	-	12.2
186		95	5	-	11.4
187		95	4	1	11.9
188		95	1	4	6.8
189		98	1	1	4.7
190	YELKIN TS	99	1	-	6.8
191		95	5	-	20.7
192		95	4	1	7.5
193		95	1	4	8.2
194		98	1	1	4.9
195	Ultralec F	99	1	-	1.9
196		95	5	-	1.4
197		95	4	1	1.7
198		95	1	4	1.8
199		98	1	1	3.3

ATPHOS 3250:  $C_{12}\text{-Ph-O-(EO)}_4\text{-P}_2\text{O}_5$ ; ATPHOS 3226:  $C_{13}\text{-Ph-O-(EO)}_6\text{-P}_2\text{O}_5$ ; Yelkin and Ultralec are lecithin based.

5 Alkyl and aryl sulfonates were also explored as surfactants for the silicone solvent. Table 10 shows the results of such materials, with and without additional water.

Table 10. Other Anionic Surfactants.

Exp	Surfactant	Surfactant Trade Name	pbw solvent	pbw surfactant	pbw Water	$\Delta E$
200	AZ6005	Witconate AOS	95	5	-	11.9
201			95	5	-	3.9
202			99	1	-	9.7
203			99	1	-	11.2
204			99	1	-	11.8
205			99	1	-	6.4
206			99	1	-	18.3
207			99	1	-	8.2
208	06417	$C_{12}\text{-SO}_4\text{Na}^+$	99	1	-	2.7
209			95	5	-	3.4
210	06206	Triton X-200	99	1	-	6.2
211			95	5	-	5.3
212			95	4	1	11.6
213			95	1	4	4.8
214			98	1	1	19.0
215	06651	DDBSA	99	1	-	51.5
216			95	5	-	50.4
217			95	4	1	52.3
218			95	1	4	47.4
219			98	1	1	49.1
220	067751	Bio-Soft D-62	95	4	1	2.9
221			95	1	4	28.4
222			98	1	1	14.3

Triton X-200:  $C_{12}\text{-Ph-O-(EO)}_3\text{-OSO}_3\text{Na}^+$ ; Witconate AOS:  $C_{14-16}\text{-SO}_4\text{Na}^+$ ; Bio-Soft D-62: Na DDBSA, 50%.

Fluoro-surfactants were also examined as shown in Table 11. Of all the varieties tried, the fluorinated quat salts and the fluoroalkyl alkoxide displayed the best performance.

5

Table 11. Fluoro-Surfactants.

Exp. No.	Surfactant Trade Name	Type	pbw solvent	pbw surfactant	pbw water	$\Delta E$
223	Fluorad FC-120	$F_{2n+1}C_n\text{SO}_3\text{NR}_4^+$	99	1	-	8.5
224	Fluorad FC-120		98	1	1	8.1
225	Fluorad FC-129	$F_{2n+1}C_n\text{COO}^-\text{K}^+$	99	1	-	1.9
226	Fluorad FC-129		98	1	1	7.4
227	Fluorad FC-135	$(F_{2n+1}C_n)_4\text{N}^+\text{T}^-$	99	1	-	13.0
228	Fluorad FC-135		98	1	1	31.9
229	Fluorad FC-170C	$F_{2n+1}C_n\text{-(EO)}_x\text{-H}$	99	1	-	10.6
230	Fluorad FC-170C		98	1	1	13.0
231	Fluorad FC-171	$F_{2n+1}C_n\text{-OR}$	99	1	-	7.5
232	Fluorad FC-171		98	1	1	21.7
233	Fluorad FC-430	$F_{2n+1}C_n\text{-COOR}$	99	1	-	10.9
234	Fluorad FC-430		98	1	1	10.4
235	Fluorad FC-740	$F_{2n+1}C_n\text{-COOR}$	99	1	-	2.2
236	Fluorad FC-740		98	1	1	3.9
237	Dynol 604	$F_{2n+1}C_n\text{SO}_2\text{N(EO)CH}_2\text{COO}^-\text{K}^+$	99	1	-	7.1
238	Dynol 604		98	1	1	1.2

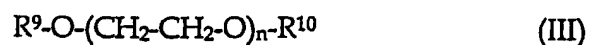
The present invention exhibits improved performance of dry cleaning agents for stain removal, particularly water soluble stains, through the addition of a surfactant, and optionally water.

## CLAIMS:

1. A dry cleaning composition, comprising a volatile cyclic, linear or branched siloxane, or combination thereof, and one or more organic surfactants.
- 5        2. The composition of Claim 1, comprising from about 90 to about 99.99 parts by weight of the volatile siloxane and from about 0.001 to less than 10 parts by weight of the surfactant or combination of surfactants.
3. The composition of Claim 2, further comprising from about 0.01 to about 15 parts by weight of water.
- 10       4. The composition of Claim 1, wherein the surfactant or surfactants are selected from the classes of nonionic, cationic, anionic and amphoteric surfactants.
5. The composition of Claim 1, wherein the organic surfactant comprises a mixture of two or more compatible surfactants of the same or  
15       different classes.
6. The composition of Claim 1, wherein the organic surfactant is a nonionic surfactant.



7. The composition of Claim 6, wherein the surfactant comprises one or more nonionic surfactants according to one or more of the structural formulas III and IV:

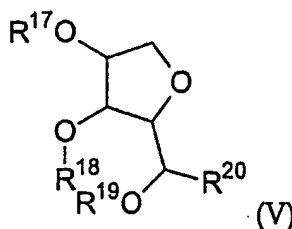


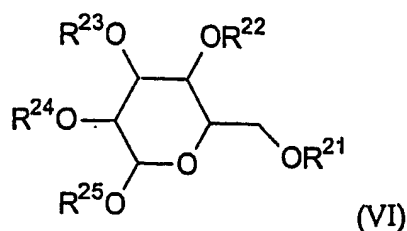
wherein:

$R^9$  is a monovalent hydrocarbon group of 1 to 30 carbons selected from the group consisting of linear, cyclic, branched, unsaturated, aromatic and fluoro containing hydrocarbon radicals,  $R^{10}$  is hydrogen or a monovalent hydrocarbon group of 1 to 30 carbons selected from the group consisting of linear, cyclic, branched, unsaturated, aromatic and fluoro containing hydrocarbon radicals, and  $n$  is from about 1 to about 100.

8. The composition of Claim 8, wherein  $R^9$  contains from 2 to about 24 carbons,  $R^{10}$  is H and  $n$  is from about 2 to about 20.

15 9. The composition of Claim 6, wherein the surfactant comprises one or more nonionic surfactants according to one or more of the structural formulas V and VI:





wherein:

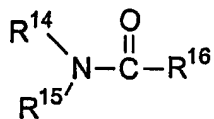
each  $R^{17}$ ,  $R^{18}$ ,  $R^{19}$ ,  $R^{20}$ ,  $R^{21}$ ,  $R^{22}$ ,  $R^{23}$ ,  $R^{24}$  and  $R^{25}$  is independently H or a monovalent hydrocarbon group of 1 to 30 carbons selected from the group consisting of linear, cyclic, and branched hydrocarbon radicals and linear, cyclic and branched chalcogen containing hydrocarbon radicals.

10. The composition of Claim 9, wherein  $R^{17}$ ,  $R^{18}$ ,  $R^{19}$ ,  $R^{20}$ ,  $R^{21}$ ,  $R^{22}$ ,  $R^{23}$ ,  $R^{24}$  and  $R^{25}$  are each H or a hydrocarbon group of 1 to 24 carbons.

11. The composition of Claim 10, wherein one of  $R^{17}$  and  $R^{20}$  is a hydrocarbon of from 8 to 24 carbons while the other is H or a hydrocarbon of from 1 to 4 carbons, such as  $-\text{CH}_2\text{OH}$  or  $-\text{CH}_2\text{CH}_3$ , and one of  $R^{21}$  and  $R^{25}$  is H or a hydrocarbon of from 8 to 24 carbons while the other is a hydrocarbon of from 1 to 4 carbons, such as  $-\text{CH}_2\text{OH}$  or  $-\text{CH}_2\text{CH}_3$ .

12. The composition of Claim 11, wherein the nonionic surfactant or surfactants are chosen from sucrose esters, glucose esters, monoglycerides, polysaccharide ethers and sorbitan-based surfactants.

13. The composition of Claim 6, wherein the surfactant comprises one or more nonionic surfactants selected from the group consisting of an amine-based surfactant and a phosphate ester-based surfactant according to the structural formulas VII and VIII:



(VII)

(VIII)

wherein:

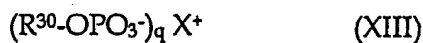
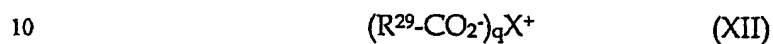
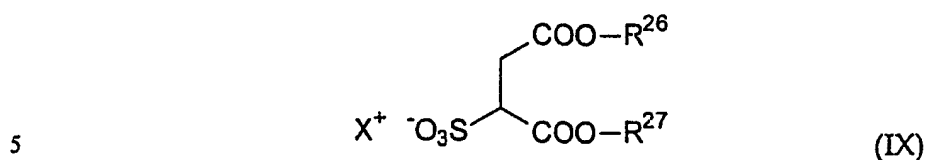
each  $\text{R}^{11}$ ,  $\text{R}^{12}$ ,  $\text{R}^{13}$ ,  $\text{R}^{14}$ ,  $\text{R}^{15}$ , and  $\text{R}^{16}$  is independently H or a monovalent hydrocarbon group of 1 to 30 carbons selected from the group consisting of linear, cyclic, branched, unsaturated, aromatic, and fluoro containing hydrocarbon radicals and chalcogen containing hydrocarbon radicals.

14. The composition of Claim 13, wherein two of  $\text{R}^{11}$ ,  $\text{R}^{12}$  and  $\text{R}^{13}$ , are H or hydrocarbon groups of 1 to 4 carbons, and one is a hydrocarbon group of from 8 to 24 carbons, and  $\text{R}^{14}$  and  $\text{R}^{15}$  are either H or hydrocarbon groups of from 1 to 4 carbons while  $\text{R}^{16}$  is a hydrocarbon group of from 8 to 24 carbons, or  $\text{R}^{14}$  and  $\text{R}^{15}$  are hydrocarbon groups of from 8 to 24 carbons while  $\text{R}^{16}$  is a hydrocarbon group of from 1 to 4 carbons.

15. The composition of Claim 14, wherein the nonionic surfactant or surfactants are chosen from alkanol amides, amine oxides, ethoxylated amines, ethoxylated amides and phosphate esters.

16. The composition of Claim 1, wherein the organic surfactant is an anionic surfactant.

17. The composition of Claim 16, wherein the surfactant comprises one or more anionic surfactants selected from the group consisting of a sulfosuccinate, sulfate, sulfonate, carboxylate, or phosphorous containing surfactant according to the structural formulas IX to XIII:



wherein:

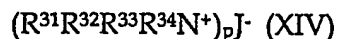
each  $\text{R}^{26}$ ,  $\text{R}^{27}$ ,  $\text{R}^{28}$ ,  $\text{R}^{29}$  and  $\text{R}^{30}$  is independently H or a monovalent hydrocarbon group of 1 to 30 carbons selected from the group consisting of linear, cyclic, and branched hydrocarbon radicals and linear, cyclic and branched chalcogen containing hydrocarbon radicals, and X is a cation selected from the group consisting of H, alkali metals, alkaline earth metals or chalcogen containing cations wherein q is the oxidation state or valence of X and whereby X does not unduly interfere with the functioning of the molecule as a surfactant.

18. The composition of Claim 17, wherein  $R^{26}$  and  $R^{27}$  are linear hydrocarbon groups of from 4 to 20 carbons,  $R^{28}$  is a hydrocarbon group of from 6 to 20 carbons,  $R^{29}$  is a hydrocarbon group of from 8 to 26 carbons, and  $R^{30}$  is a hydrocarbon of from 8 to 30 carbons.

5 19. The composition of Claim 18, wherein  $R^{26}$  and  $R^{27}$  are linear hydrocarbon groups of from 6 to 13 carbons,  $R^{28}$  is a hydrocarbon group of from 8 to 16 carbons, and  $R^{29}$  is a hydrocarbon group of from 10 to 20 carbons.

20. The composition of Claim 1, wherein the organic surfactant is a cationic surfactant.

10 21. The composition of Claim 20, wherein the surfactant comprises one or more cationic surfactants that may be a quaternary amine-based surfactant according to the structural formula XIV:



wherein:

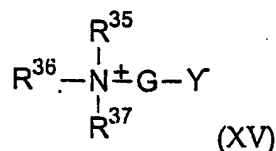
15 each  $R^{31}$ ,  $R^{32}$ ,  $R^{33}$ , and  $R^{34}$  is independently H or a monovalent hydrocarbon group of 1 to 30 carbons selected from the group consisting of linear, cyclic, and branched hydrocarbon radicals and linear, cyclic and branched chalcogen containing hydrocarbon radicals, and j is a suitable anion having a valence or oxidation state p that does not unduly interfere with the functioning of the  
20 molecule as a surfactant.

22. The composition of Claim 21, wherein  $R^{31}$  and  $R^{32}$  are hydrocarbon groups of from 1 to 4 carbons, and  $R^{33}$  and  $R^{34}$  are hydrocarbon groups of from 6 to 30 carbons.

23. The composition of Claim 22, wherein  $R^{31}$  and  $R^{32}$  are methyl,  
25 and  $R^{33}$  and  $R^{34}$  are hydrocarbon groups of from 8 to 24 carbons.

24. The composition of Claim 1, wherein the organic surfactant is an amphoteric surfactant.

25. The composition of Claim 24, wherein the surfactant comprises one or more amphoteric surfactants according to the structural formula XV:



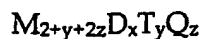
5

wherein:

each  $\text{R}^{35}$ ,  $\text{R}^{36}$  and  $\text{R}^{37}$  is independently H or a monovalent hydrocarbon group of 1 to 30 carbons selected from the group consisting of linear, cyclic, and branched hydrocarbon radicals and linear, cyclic and branched chalcogen  
10 containing hydrocarbon radicals, G is a divalent spacer group, and Y is a carboxylate, sulfonate, sulfate, phosphonate or other similar group.

26. The composition of Claim 25, wherein  $\text{R}^{35}$  is a hydrocarbon of from 1 to 4 carbons, and  $\text{R}^{36}$  and  $\text{R}^{37}$  are hydrocarbons of from 6 to 24 carbons.

27. The composition of Claim 1, wherein the linear or branched, volatile siloxane comprises one or more compounds of the structural formula:



wherein:

5 M is  $R^1_3SiO_{1/2}$ ;

D is  $R^2R^3SiO_{2/2}$ ;

T is  $R^4SiO_{3/2}$ ;

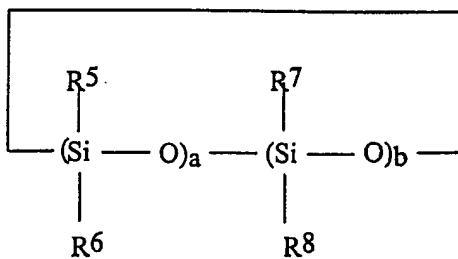
and Q is  $SiO_{4/2}$

$R^1$ ,  $R^2$ ,  $R^3$  and  $R^4$  are each independently a monovalent hydrocarbon radical;

10 and

x and y are each integers, wherein  $0 \leq x \leq 10$  and  $0 \leq y \leq 10$  and  $0 \leq z \leq 10$ .

28. The composition of Claim 1, wherein the cyclic siloxane component comprises one or more compounds of the structural formula:



15

wherein:

$R^5$ ,  $R^6$ ,  $R^7$  and  $R^8$  are each independently a monovalent hydrocarbon group;

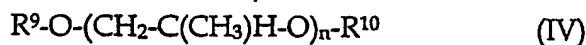
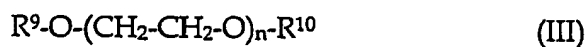
and

a and b are each integers wherein  $0 \leq a \leq 10$  and  $0 \leq b \leq 10$ , provided that  $3 \leq$

20  $(a + b) \leq 10$ .

29. A method for dry cleaning an article, comprising contacting the article with a composition comprising a cyclic, linear or branched siloxane and one or more organic surfactants selected from the classes of nonionic, cationic, anionic and amphoteric surfactants.

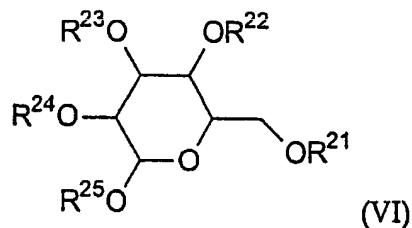
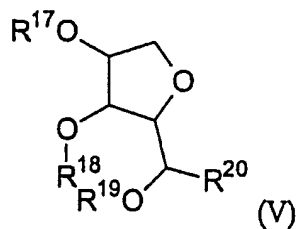
5 30. The method of Claim 29, wherein the surfactant comprises one or more nonionic surfactants according to one or more of the structural formulas III and IV:



10 wherein:

R<sup>9</sup> is a monovalent hydrocarbon group of 1 to 30 carbons that may be linear, cyclic, branched, unsaturated, aromatic or fluoro containing, R<sup>10</sup> is hydrogen or a monovalent hydrocarbon group of 1 to 30 carbons that may be linear, cyclic, branched, unsaturated, aromatic or fluoro containing, and n is  
15 from about 1 to about 100.

31. The method of Claim 29, wherein the surfactant comprises one or more nonionic surfactants according to one or more of the structural formulas V and VI:



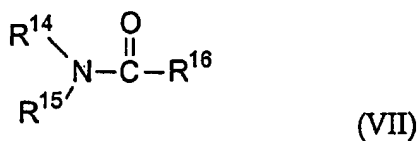
20



wherein:

each R<sup>17</sup>, R<sup>18</sup>, R<sup>19</sup>, R<sup>20</sup>, R<sup>21</sup>, R<sup>22</sup>, R<sup>23</sup>, R<sup>24</sup> and R<sup>25</sup> is independently H or a monovalent hydrocarbon group of 1 to 30 carbons selected from the group consisting of linear, cyclic, and branched hydrocarbon radicals and linear, cyclic and branched chalcogen containing hydrocarbon radicals.

32. The method of Claim 29, wherein the surfactant comprises one or more nonionic surfactants that may be an amine-based or phosphate ester-based surfactant according to one or more of the structural formulas VII and VIII:

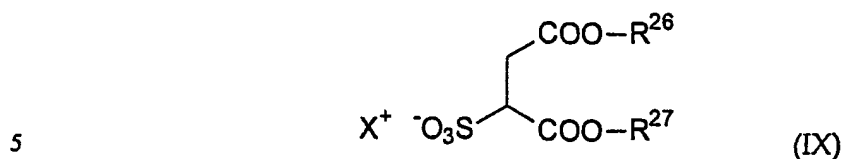


(VIII)

wherein:

each R<sup>11</sup>, R<sup>12</sup>, R<sup>13</sup>, R<sup>14</sup>, R<sup>15</sup>, and R<sup>16</sup> is independently H or a monovalent hydrocarbon group of 1 to 30 carbons selected from the group consisting of linear, cyclic, and branched hydrocarbon radicals and linear, cyclic and branched chalcogen containing hydrocarbon radicals.

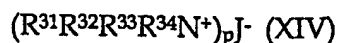
33. The method of Claim 29, wherein the surfactant comprises one or more anionic surfactants that may be a sulfosuccinate, sulfate, sulfonate, carboxylate, or phosphorous containing surfactant according to one or more of the structural formulas IX to XIII:



10 wherein:

each  $\text{R}^{26}$ ,  $\text{R}^{27}$ ,  $\text{R}^{28}$ ,  $\text{R}^{29}$  and  $\text{R}^{30}$  is independently H or a monovalent hydrocarbon group of 1 to 30 carbons selected from the group consisting of linear, cyclic, and branched hydrocarbon radicals and linear, cyclic and branched chalcogen containing hydrocarbon radicals, and X is a cation  
15 selected from the group consisting of H, alkali metals, alkaline earth metals or chalcogen containing cations wherein q is the oxidation state or valence of X and whereby X does not unduly interfere with the functioning of the molecule as a surfactant..

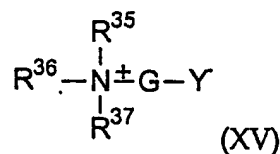
34. The method of Claim 29, wherein the surfactant comprises one or more cationic surfactants that may be a quaternary amine-based surfactant according to the structural formula XIV:



5 wherein:

each  $R^{31}$ ,  $R^{32}$ ,  $R^{33}$ , and  $R^{34}$  is independently H or a monovalent hydrocarbon group of 1 to 30 carbons selected from the group consisting of linear, cyclic, and branched hydrocarbon radicals and linear, cyclic and branched chalcogen containing hydrocarbon radicals, and J is a suitable anion having a valence or  
10 oxidation state p that does not unduly interfere with the functioning of the molecule as a surfactant.

35. The method of Claim 29, wherein the surfactant comprises one or more amphoteric surfactants according to the structural formula XV:



15 wherein:

each  $R^{35}$ ,  $R^{36}$  and  $R^{37}$  is independently H or a monovalent hydrocarbon group of 1 to 30 carbons selected from the group consisting of linear, cyclic, and branched hydrocarbon radicals and linear, cyclic and branched chalcogen containing hydrocarbon radicals, G is a divalent spacer group, and Y is a  
20 carboxylate, sulfonate, sulfate, phosphonate or other similar group.

## INTERNATIONAL SEARCH REPORT

International Application No

PCT/US 01/26918

A. CLASSIFICATION OF SUBJECT MATTER  
IPC 7 D06L1/04

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 7 D06L C11D

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

WPI Data, EPO-Internal, PAJ

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☒ Further documents are listed in the continuation of box C.☒ Patent family members are listed in annex.

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Date of the actual completion of the international search

12 March 2002

Date of mailing of the international search report

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	page 3, line 27 - line 31; claims 7-12 -----	
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	page 7, paragraph 5 -page 8, paragraph 1 page 20, paragraph 2 -page 23, paragraph 2; claims 7-12 -----	
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